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Surface Analysis of Bearings

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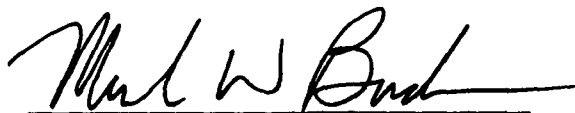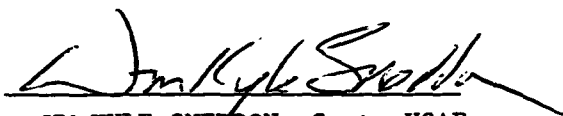El Segundo, California

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The report reviews the techniques used to conduct a failure analysis of precision bearings. The review is arranged in a manner consistent with the sequence of bearing disassembly. The information to be gained by and the limitations of each technique are identified, and appropriate examples of fluid- and solid-lubricated systems are presented. The techniques used for lubricant analysis and micro-examination are reviewed, but particular emphasis is given to their integration with surface analysis.

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PREFACE

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1. INTRODUCTION

Precision ball bearings used in gyroscopes and in spacecraft pointing mechanisms critically depend upon superior lubricant performance in order to function properly. The lubricants are either solid, liquid, or grease, with the latter two usually containing boundary or extreme pressure additives. These additives modify the bearing surface composition, which reduces wear, i.e., provides lubrication during low-speed operation. These mechanisms generally contain the optimum amount of lubricant needed to minimize torque — either insufficient or excessive quantities of lubricant lead to an increase in torque. Thus, bearing failure for these mechanisms generally occurs because the lubricant becomes inadequate, either through chemical degradation, evaporative loss, or migration of the lubricant away from the contact zone. In contrast, bearing failure by classical fatigue occurs in only 10% of steel aerospace bearings and is virtually nonexistent in spacecraft bearings.[1,2] Therefore, the failure analyses of these precision bearings focus on the determination of lubricant failure and the measurement of bearing wear.

This report reviews the techniques used to conduct a failure analysis of precision bearings. The review is arranged in a manner consistent with the sequence of bearing disassembly. The information to be gained by and the limitations of each technique will be identified, and appropriate examples will be presented. The reader should note that a multi-technique approach is critical to determining the cause of bearing failure. Hopple has written an excellent guide to the problem with regard to lubricant analysis and micro-examination.[3] The present text reviews those techniques, but also emphasizes their integration with surface analysis. Further details of the surface analysis techniques can be found elsewhere in the literature.[4]

2. DISASSEMBLY

2.1 EXAMINATION, OPTICAL MICROSCOPY, PHOTOGRAPHY

Physical examination of the bearing provides the initial indication of the failure mode and in some cases can suggest a particular emphasis for the application of subsequent techniques. The following should be noted by low-power (10 - 100 X) optical microscopy of the bearing: condition of lubricant (e.g., discolored, dry, presence or absence), presence and nature of debris, retainer condition (e.g., fractured, discolored), and any other features of interest. Photography provides a visual record of the bearing condition. Photographs of the disassembled bearing can also act as a guide to direct the location of further analyses.

Most bearings can be disassembled using conventional methods. For less accessible components that are cylindrical in shape, disassembly using a jeweler's lathe is preferred. Lathe turnings, unlike grinding or sawing debris, are easily distinguishable from most forms of bearing wear or contamination particles.[3]

2.2 GAS ANALYSIS BY MASS SPECTROMETRY

Some mechanisms, particularly gyroscopes, have bearings enclosed in hermetically sealed atmospheres to prevent contamination. The sealed atmosphere can be sampled and analyzed by mass spectrometry (Residual Gas Analyzer) to verify the integrity of the seal and to detect impurities.

For space applications, many bearing qualification tests are conducted in vacuum to simulate the operational environment. A mass spectrometer can be attached to the vacuum test chamber to ensure that a clean vacuum (e.g., no leaks from atmosphere, no cleaning solvents present, etc.) is maintained during the test.

2.3 LUBRICANT ANALYSIS AND REMOVAL

When bearings are lubricated by solids, such as MoS_2 or polymers, these lubricant films can be analyzed along with the bearing surfaces as discussed in Sections 3 and 4. However, liquid lubricants and the liquid portions of greases are not amenable to those techniques. Special techniques must be employed to analyze these lubricants prior to micro-examination and surface analysis of the bearings. Indeed, the liquids must be removed prior to bearing analysis, and the removal needs to be executed in a manner that minimizes disturbance to the bearing surface. Greases, which consist of liquid lubricant and thickener (often a soap compound) components, require special procedures for separation and independent analyses of these components. Detailed procedures for lubricant analyses have been reported by Carré[5] and Hopple,[3] but aspects will be briefly reviewed here.

Fluid lubricants are analyzed for the following reasons: (1) to verify that the appropriate (specified) lubricant was used; (2) to assess chemical degradation (e.g., polymerization, fragmentation, oxidation) of the lubricant; (3) to determine if evaporative loss of the lighter molecular weight fractions has occurred; (4) to detect fluid contamination; (5) to select an appropriate sol-

vent for bearing cleaning prior to surface analysis; and (6) to determine the extent of grease separation (oil loss).

Two common techniques for lubricant analysis are infrared spectroscopy (IR) and gas chromatography (GC). IR measures the vibrational modes of organic molecules, which can be used to assess functional groups (e.g., alcohols, ketones, etc.) of the lubricant. GC separates the lubricant molecules by boiling point, providing an assessment of the molecular weight distribution, which in part determines viscosity, of the liquid lubricant. In some GC systems, special columns can be used to separate functional groups. If the GC is combined with a mass spectrometer (GC-MS), the molecular structure of the separated molecules can be further assessed. In all cases, careful comparison of data from bearing lubricant samples and from lubricant and other standards is required to meet the objectives mentioned in the previous paragraph.

Solid debris particles may be present in the liquid and can be collected by filtration and investigated with the techniques of Sections 3 and 4. The thickener component of greases can be preserved by freeze-drying, and its structure examined by microscopy. For example, overheating of a grease can lead to melting of the thickener, which causes the porous structure to collapse, rendering it unable to retain sufficient oil.[3]

Prior to microscopy and surface analysis in vacuum, liquid lubricants have to be removed. Heptane, which because it is used as a standard for GCs can be obtained readily in high purity, is often used to remove hydrocarbon liquids, while Freon is utilized to remove fluorocarbon liquids. Although ultrasonic agitation is generally used, we try to limit the duration to the minimum time sufficient to remove the liquid without further mechanically disturbing remnant additive-derived surface films. For this reason, bearing balls are often separated to prevent impingement by rolling during cleaning.

3. MICRO-EXAMINATION

3.1 SCANNING ELECTRON MICROSCOPY

Scanning electron microscopy (SEM) is one of the more powerful tools for bearing failure analysis. SEM provides direct assessment of the bearing surface morphology so that wear can be identified. Excellent texts of SEM studies of wear and fatigue exist and should be consulted.[6,7] In the present context, SEM also is very useful in studying remnant lubricant films, remnant additive films, debris particles, or the freeze-dried thickener components of greases.[8] SEM provides structural information on the morphology of these surface regions. In combination with X-ray spectroscopy, either using energy-dispersive (EDS) or wavelength-dispersive (WDS) detectors, SEM can provide the spatially resolved composition distribution of elements present near (0.5 - 1 μm depth) the surface.

A key advantage in using SEM to complement the surface analysis techniques in Section 4 is that SEM, because of high signal collection rates and less-stringent vacuum requirements, enables more rapid analysis of bearing surfaces compared to the ultra-high vacuum (UHV) techniques. SEM is used to survey the various types of surface regions present on bearing surfaces. Representative regions are then selected for further, more time-consuming, surface analysis. SEM also has better spatial resolution than the surface spectroscopies. SEMs that can produce electron beam diameters (spot size) of 4 to 6 nm are now available.

In examining bearing surfaces, multiple-mode images (i.e., secondary electron [SE] vs. backscattered electron [BSE] and high-energy vs. low-energy imaging modes) of surface regions are particularly informative. The contrast in SE images is strongly dependent upon surface topography relative to the detector but is relatively insensitive to the composition (atomic number [Z]) of the irradiated region. BSE images are strongly dependent upon atomic number, as well as topography, with higher Z elements generating more backscattered electrons. Lower primary-beam energy in BSE mode enhances the surface sensitivity of the image, emphasizing elements at the surface, although spatial resolution degrades because the electron spot size increases as beam energy decreases.

For example, bearing raceways, operated under low-speed oscillatory conditions and lubricated with a synthetic hydrocarbon oil, a multiply-alkylated cyclopentane (MAC), and the extreme pressure additive lead naphthenate (Pbnp) were studied by SEM. BSE mode at 5 keV revealed the presence of a bright overlayer that was less evident at 20 keV and not evident in SE mode, as shown in Figure 1. Surface analysis (Section 4) revealed that the bright regions contained Pb and C decomposed from Pbnp. The high atomic number of Pb (82) caused the bright contrast in BSE mode, relative to Fe (26) or C (6). These Pb-containing films provided protection against microdeformation and wear that occurred in tests of the oil without Pbnp additive, as shown in Figure 2.

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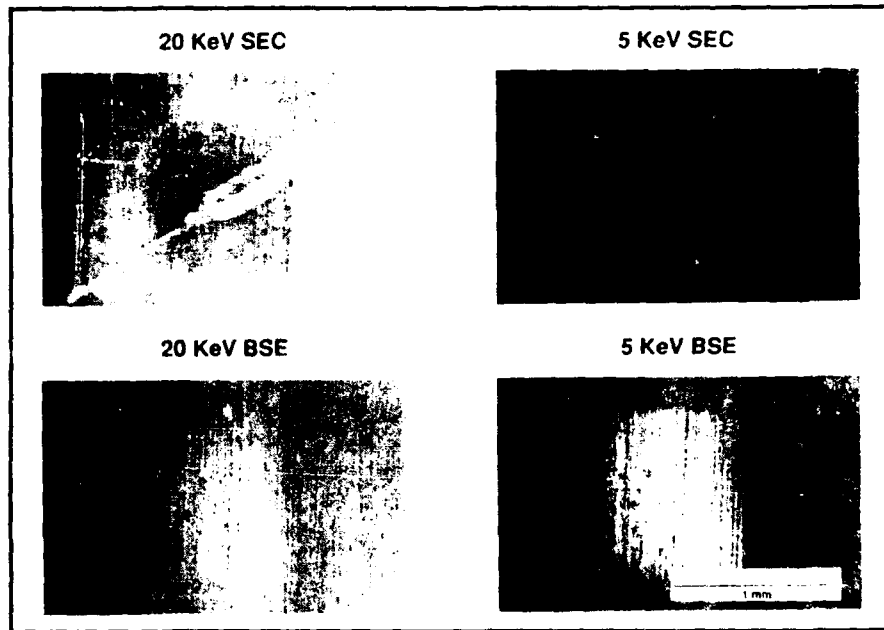
Figure 1. SEM photographs showing a 440C steel bearing raceway lu! .icated with MAC oil and lead naphthenate (Pbnp). A Pb-containing overlayer film in the contact region is very evident (i.e., has a very bright appearance) using BSE mode, particularly at 5 keV, which is more surface sensitive.

Figure 2. SEM micrographs (20 keV BSE) comparing two bearing raceways lubricated with a MAC oil. One raceway had Pbnp present, as in Figure 1, while the other raceway was lubricated with only the oil. Deformation, evident with the pure oil, is avoided when the Pbnp additive is present. The SEM micrographs provide information on the bearing and lubricant overlayer surfaces.

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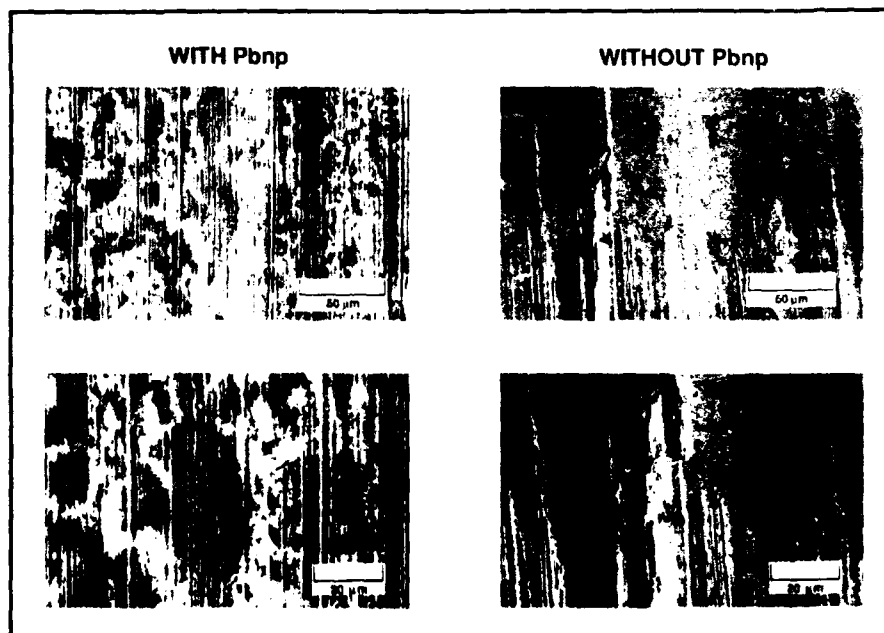The BSE mode of SEM was also useful in studying the wear and deformation of sputter-deposited MoS_2 solid-lubricant films on thrust bearings operated unidirectionally. Figure 3 shows a bearing after failure (defined as unacceptable motor torque). A 1-μm film was initially present on the bearing. The micrographs show the pathway of ball contact where most of the film has been removed, as confirmed by EDS and surface analysis (Section 4).

Although the BSE mode can be useful in spatially resolving different compositions, there are limitations. Topography changes also affect contrast in BSE. Correlation of BSE gray levels with different chemical phases has to be continually verified by EDS/WDS, or by surface analysis, during investigation. New samples require special scrutiny to confirm previous trends. Contrast in the BSE mode is very system dependent. Although Pb yields strong contrast in BSE, P from the additive tricresyl phosphate (TCP) does not yield a strong contrast. BSE imaging provides little benefit in that case.
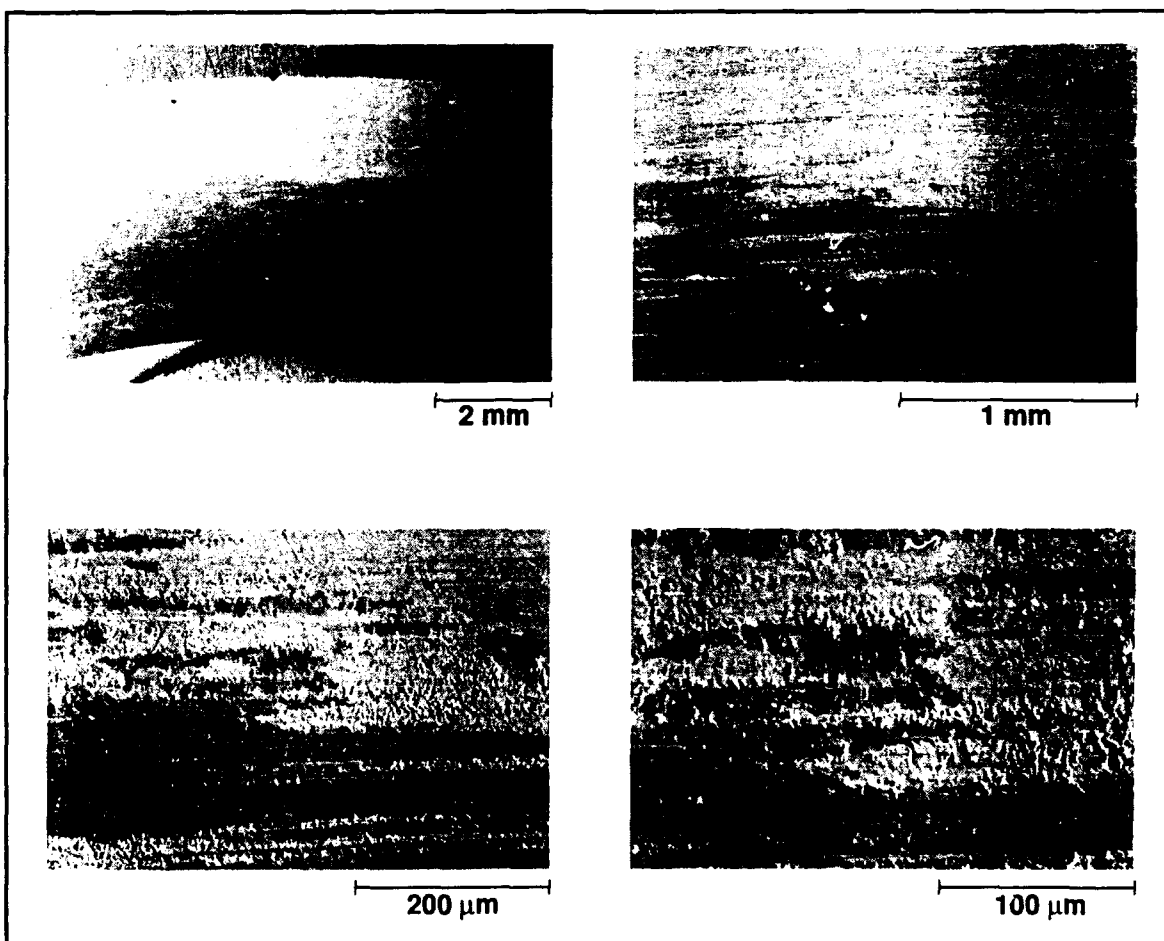
Figure 3. SEM micrographs of a 440C steel thrust bearing raceway lubricated with sputter-deposited MoS_2 (1 μm thick) after testing and failure, defined as a significant torque rise. The BSE mode reveals that the film has been significantly removed from the contact region, appearing as light-gray and dark-gray areas, respectively, in these BSE micrographs. Note the presence of carbides in the region where film has been removed.

3.2 PROFILOMETRY

Profilometry by stylus methods is used to measure bearing surface roughness and curvature. Use of this technique can determine if wear or additive film buildup has occurred in contact regions. The wear or redistribution of solid lubricants in bearings can be studied. (Initial solid-lubricant film thickness prior to testing can also be measured if sharp steps are present between coated and uncoated regions. This is usually done on witness plates as opposed to bearings.) The stylus tip may deform the soft solid lubricant or additive film and yield an erroneously low thickness. If the stylus deformation width on the film is examined and measured by SEM, the depression and correct thickness can be calculated because the tip geometry is known.[9]

4. SURFACE ANALYSIS

4.1 AUGER ELECTRON SPECTROSCOPY

Auger electron spectroscopy (AES), often executed in a scanning Auger microprobe (SAM), is used in the analysis of bearings to determine the elemental composition of the near-surface ($\leq$ 10 nm) region, including overlayers, contaminants, and exposed bearing material. If argon ion sputtering is used to remove material, compositional depth profiles can be obtained, which can help to determine the thickness of overlayers and contaminants. Because the electron beam used to generate Auger electrons can be focused and scanned, in principle, down to a beam size of 30 nm, spatially resolved maps of surface composition can be generated. In practice, SAM is used after SEM investigation has identified representative regions for analysis. [The electron beam of the SEM might, in some cases, damage a carbonaceous surface layer or induce contamination upon the surface. The investigator might elect to examine some samples directly with AES (or XPS) after optical examination.] The SAM, when detecting secondary electrons, can be operated as a SEM, although only the most recent instruments have beam sizes, and therefore spatial resolution, comparable to standard SEMs. The minimum detection limit for most elements by AES is 0.1 - 1 atomic %. Older AES systems, of which there are many still in use, display peak derivatives because lock-in amplifiers are used in the detection scheme. The derivative peak height is normally related to the quantity of element present in the detected volume, after normalization and cross-section sensitivities are taken into account. However, overlap of adjacent derivative peaks or shape changes due to variations in elemental bonding (i.e., oxidation) can distort this direct relationship, making quantitative analysis less certain in those cases. Newer AES systems avoid this problem by collecting data in summation mode.

Figure 4 shows profiles, with a corresponding SEM image, from a raceway lubricated with Pbnp and MAC oil as discussed in Section 3.1. The ball contact region had two types of additive films visible in BSE mode: a bright material with AES depth profile (labeled "T") and a dark material with AES profiles for thick and thin regions (labeled "C" and "B", respectively). The AES data show that the bright material contains 12 to 18% Pb with carbon and oxygen while the dark regions contain only 3 to 5% Pb with more carbon. The higher Pb composition of the film represented in T yields bright BSE images while the relatively high carbon content, and lower Pb content, causes dark images to form in regions C and B, relative to the gray appearance of most of the steel. This result is entirely consistent with the atomic numbers of these elements as discussed in Section 3.1. These films are decomposition products of Pbnp — region T is more decomposed (i.e., has a higher Pb content) than regions C and B. It is hypothesized that region T achieved higher temperature or stress or both, for unknown reasons, than adjacent areas. Other studies have shown that these factors promote Pbnp decomposition.[10]

Figure 5 shows AES spectra from apparently worn and unworn regions on the MoS_2-lubricated thrust bearing shown previously in Figure 3. AES confirms that the worn region has exposed steel, though there are still remnant MoS_2 particles present in the irradiated regions. Spatial resolution of the particles versus the exposed regions was not possible, apparently because the beam size of the particular SAM used in this study was greater than 3 µm.
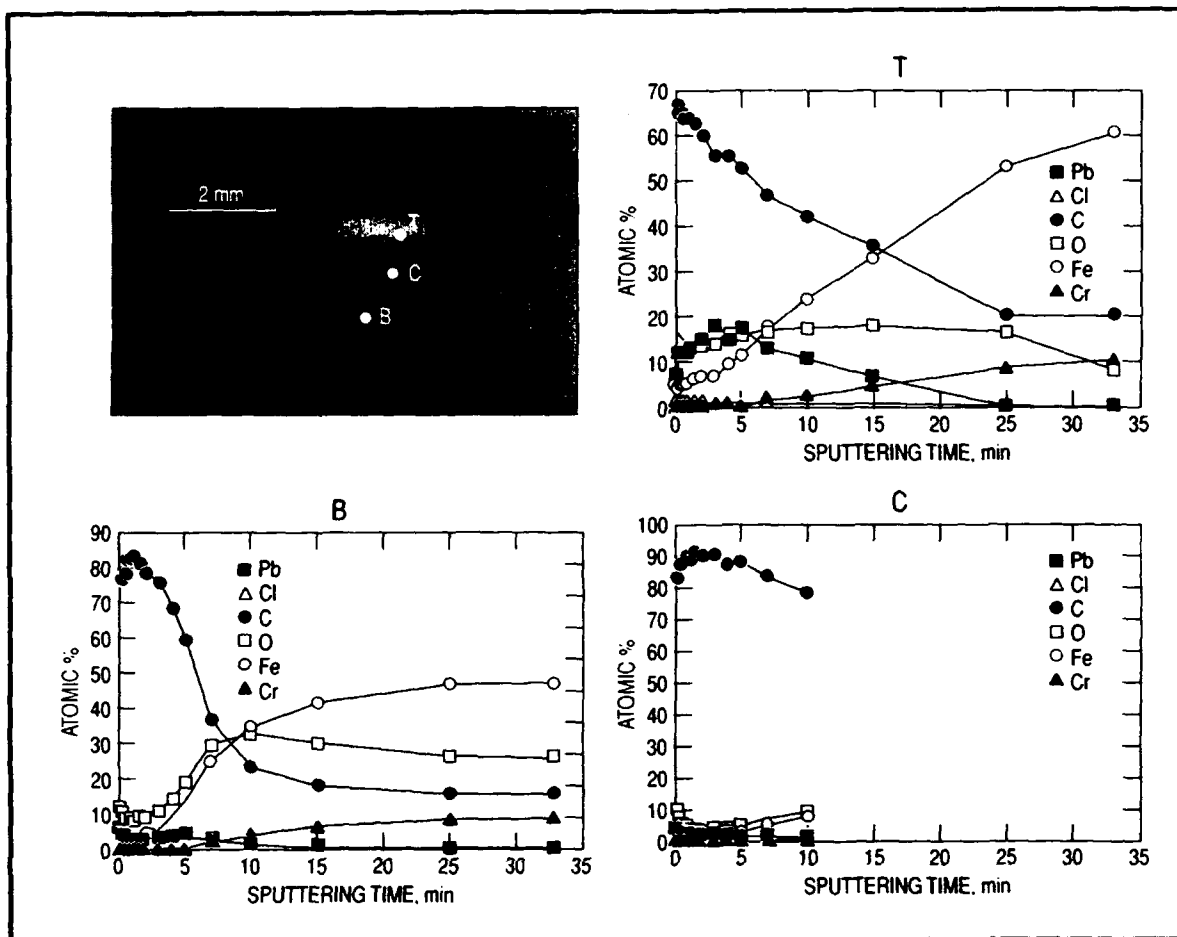
Figure 4. SEM micrograph (5 keV, BSE mode) and AES sputter-profile spectra of a raceway lubricant with MAC oil and Pbnp. Unlike Figures 1 and 2, this particular raceway revealed overlayers of variable Pbnp decomposition in the contact region. A film containing 12 to 18% Pb is present in Area "T" (similar to Figures 1 and 2), while more carbonaceous films are present in areas "C" and "B". The sputter profiling rate is estimated to be 10 nm/min. The film in Area C is very thick, and sputtering was discontinued after 10 min.

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Figure 5. AES spectra from the raceway shown in Figure 3, from an unworn and worn region. The steel substrate is evident without sputtering in the worn area, clearly showing that the MoS_2 has been removed in areas. However, MoS_2 islands or debris are still present.
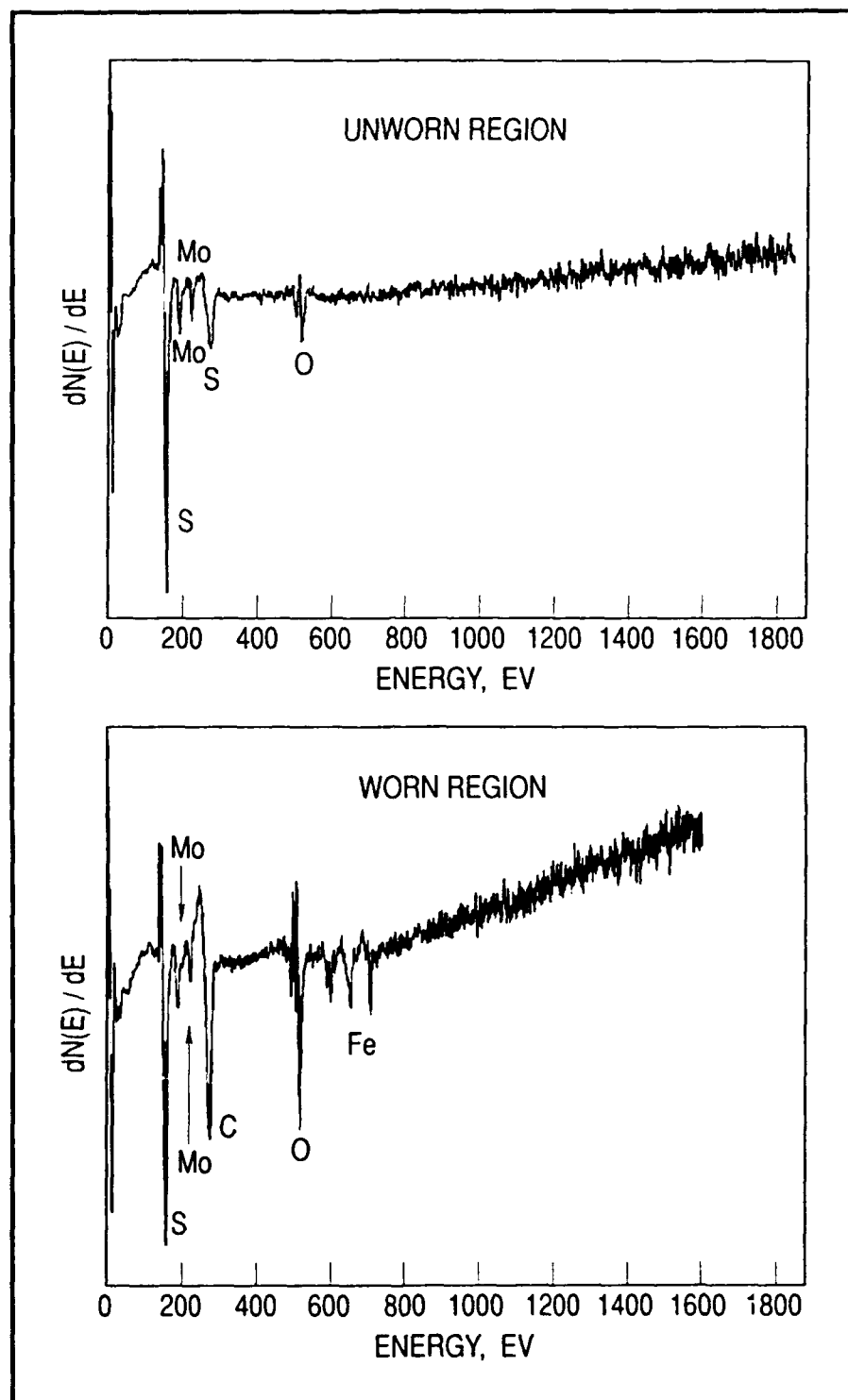
4.2 PHOTOELECTRON SPECTROSCOPY

X-ray photoelectron spectroscopy (XPS) is used to determine the chemical composition (i.e., both the presence and bonding state of elements) present in the near-surface region (< 10 nm) of a bearing. The minimum detection sensitivity of XPS is similar to AES (i.e., 1 to ~ 3 at.%) although cross-sections for particular elements are different for the two techniques. The X-ray spot size for better instruments can be < 0.5 mm. Thus, AES, and particularly SEM, have better spatial resolution than XPS. Obtaining high-resolution XPS data often takes longer than AES. While a single AES scan is usually accomplished in 2 to 5 min, only low-energy resolution XPS data can be obtained in that nominal time frame. For bearing analysis, XPS is reserved for selected samples after screening by optical microscopy and SEM. (After SEM examination, representative samples are directed to either AES or XPS since destructive sputtering is normally executed in both cases to obtain depth profiles. In cases where electron-beam-induced damage and/or contamination are likely, the investigator may choose to examine some samples with XPS immediately after optical microscopy. Comparison of these samples using XPS with other samples examined by SEM or AES prior to XPS analysis can determine if beam-induced artifacts are an issue.)

XPS of bearing surfaces can yield critical insight into the chemical mechanisms of boundary lubrication. For example, XPS has been used to examine the bearing races lubricated with the Pbnp/MAC discussed above. The XPS instrument used to examine the bearing races had a monochromatized X-ray beam with a spot size of approximately 300 μm, allowing examination both inside and outside of the contact areas. Quantitative analysis of the bearing surface composition is available with XPS. In unreacted Pbnp, XPS shows that the molecule contains approximately 3 - 4 atomic % Pb. The much higher concentrations of Pb observed by both XPS and AES (~ 20%) in some bearing contact areas conclusively indicate that the Pbnp is breaking down in the contact region and that much of the hydrocarbon residue is washed away. Typically, Pb concentrations in areas outside of the contact region are closer to the 3% expected for unreacted Pbnp.

The sensitivity of XPS to chemical state is demonstrated in Figure 6, where the strongest peaks associated with C and Pb from Pbnp, obtained both inside and outside of a bearing contact scar, are presented. The C data show at least two principal peaks of interest, one near 285 eV associated with hydrocarbon species and one near 289 eV, indicating C bonded to the more highly electronegative O atoms in the carboxylate group of the naphthenate chain. One method of tracking the reactivity of the Pbnp is to follow the XPS intensity of the surface Pb relative to the carboxylate peak. Specifically, Figure 6 data obtained within a high-lead-containing wear scar show a much lower carboxylate peak intensity relative to the amount of surface lead than areas outside of the contact area. Such changes would be expected if the Pbnp was reacting with the bearing surface by losing a naphthenate chain. Ideally, chemical-state information regarding the Pb could also be obtained directly from the binding energy positions of the XPS peaks. However, the effects of the environment (specifically, oxidation of the Pb and/or contamination) both during testing and in sample handling prior to analysis can cloud such results. Care must be taken to ensure that environmental effects on chemical state information have been considered in any surface analysis.

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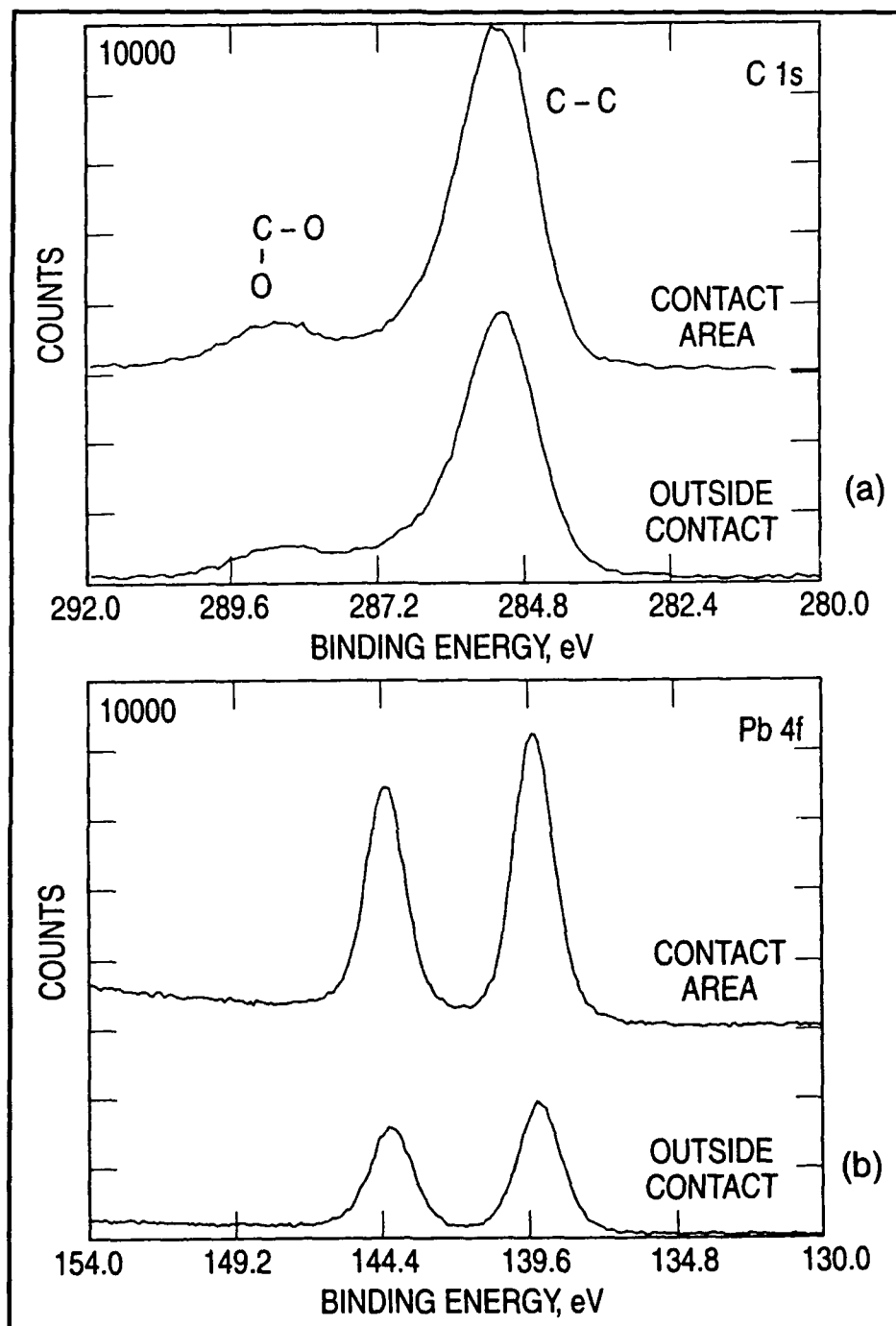Figure 6. The XPS (a) C 1s peaks and (b) Pb 4f$_{7/2,5/2}$ doublet obtained from a Pbnp/MAC-lubricated bearing race run under oscillatory conditions. The data were obtained both inside and outside of the visible contact area on the race as indicated on the diagram. The intensity ratios of the Pb 4f$_{7/2}$ peak to the carboxylate C peak are approximately 6:1 inside the contact area and 3:1 outside the contact area.

The surface chemical interactions occurring in a lubricated bearing running under boundary conditions are normally so complex that a comparison to model surfaces prepared under more controlled conditions is usually beneficial. The chemistry of Pbnp on model steel surfaces has been studied with XPS and compared to the chemistry occurring in the ball-race contacts just described.[10,11] The spectra in Figure 7 show the C and Pb peaks from a steel coupon immersed in a solution containing Pbnp; a region of the coupon was scratched with a diamond scribe while submerged to simulate extreme wear. The ratio of the surface Pb to the carboxylate C peak intensity in the unscratched area of the model surface was very similar to the unworn area of the bearing race, while the data obtained from the scratched area of the coupon more closely resembled the bearing contact area. The coupon experiments also followed the chemical reactions occurring on the steel surface in more detail than is possible for a real bearing. Use of coupons allowed XPS examination of peak changes occurring at high temperature in order to model the extreme conditions occurring during the boundary wear.[11] These high-temperature experiments indicate that the metallic iron present in steel bearings can chemically reduce Pbnp to metallic Pb (which can behave as a solid lubricant), a reaction which may explain the high lead concentrations found in the bearing wear scars and the boundary protection afforded by Pbnp.

The very high cross section for Pb in XPS makes XPS an extremely useful technique for the study of Pbnp reactivity on steels. In contrast, following the chemistry of the additive TCP by studying the P XPS signal is much more difficult because XPS is roughly 20 times less sensitive to P than to Pb. However, P can be observed on bearing surfaces treated with TCP or run with TCP-containing lubricants. Although much more analysis time is needed to obtain chemical state information than for Pb, the XPS data indicate that the P remains in a phosphate-like bonding moiety upon adsorption to steel. Possible chemical reactions that might occur during bearing operation are currently being studied. Generally, it is extremely important to understand the strengths and limitations of XPS in studying bearing surface chemistry before embarking on such an experiment and during analyses of the data obtained in such an experiment.

XPS has been used very little, to date, to study sputter-deposited MoS_2 films on actual bearings due to the limited spatial resolution of the technique and the difficulty of examining curved surfaces. XPS has been used quite successfully in examining witness plates used in deposition runs of MoS_2. A recent investigation studied the oxygen in these films, stemming from the deposition process and from post-deposition sources.[12] In particular, phases of $MoS_{2-x}O_x$ containing substitutional oxygen were identified.

4.3 SIMS

An alternative to AES in obtaining elemental information is to use ion scattering techniques such as secondary ion mass spectrometry (SIMS)[13] or secondary neutral mass spectrometry (SNMS).[14] The major advantage of these techniques is better sensitivity: the sensitivity of SIMS ranges from 10^{-6} to 0.1 at.%, depending upon the element; SNMS is generally 10^{-3} at.%; AES is 0.1 to 1 at.%. The spatial resolution of SIMS/SNMS is not as good as AES. These techniques are inherently destructive since sputtering of the surface is required for data collection.
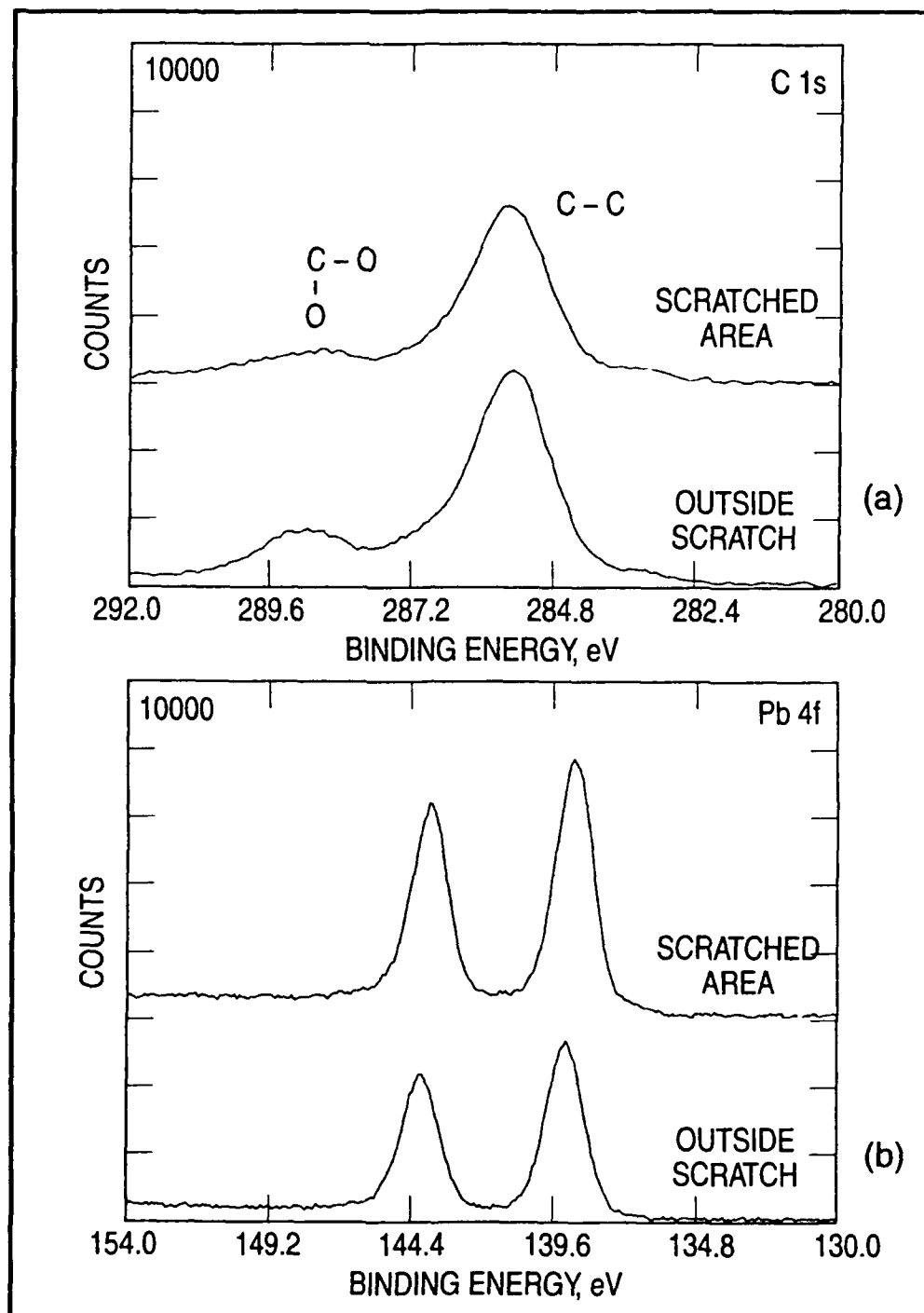
Figure 7. The XPS (a) C 1s peaks and (b) Pb 4f$_{7/2,5/2}$ doublet from a Pbnp-treated 440C stainless steel surface. The data were obtained from an undisturbed portion of the surface and from an area scratched during the Pbnp immersion treatment. The Pb:carboxylate peak ratios in the scratched areas are very similar to those observed in the contact region of the bearing in Figure 6.

4.4 VIBRATIONAL SPECTROSCOPY

Vibrational spectroscopies can be used to obtain information about organic residues on bearing surfaces. In particular, Fourier-transform infrared (FTIR) microscopes can provide spatially resolved structural information on overlayer films. The technique does not, however, have the surface sensitivity of AES, XPS, or SIMS/SNMS. High-resolution electron energy loss spectroscopy (HREELS) does provide surface-sensitive vibrational information, which is particularly useful in elucidating the structure of thin organic molecular layers. However, HREELS does not appear to be in use for bearing analysis at this time. Investigators using this technique seem to be using small samples that provide model surfaces for surface science research.

5. FUTURE DIRECTIONS

Improved equipment of established techniques and new techniques are continually being introduced for the surface analysis of bearings. High spatial resolution SAMs are becoming available that are effectively UHV SEMs as well as SAMs. Scanning probe microscopies, such as scanning tunneling microscopy (STM) and atomic force microscopy (AFM), are becoming widely used in tribology research. AFMs designed to measure normal and lateral forces can assess the local frictional properties of surfaces. STM/AFM have not been used significantly in bearing analysis. These microscopies can provide, at a minimum, atomic resolution structural information and profilometry, and their use should increase to complement conventional profilometry. It would be very beneficial and convenient for bearing analysis to have a STM or AFM integrated into a SEM or in a high spatial resolution SAM. The SEM/SAM could view the bearing over a wide range of magnifications, while the STM/AFM could be dropped onto selected small regions for detailed investigation.

The use of XPS to study bearing surfaces has been limited by several factors including X-ray spot size, fairly lengthy data accumulation periods, and inflexibility in sample handling procedures. Improvements are continually being made in generating smaller-diameter X-ray beams that are still capable of providing a sufficient signal-to-noise ratio for data collection. These smaller analysis areas should allow the study of smaller wear features in bearings. In addition, techniques such as photoelectron microscopy are currently being developed that should allow detailed chemical mapping of surfaces of interest. The lengthy data collection times have been considerably shortened with multi-channel detectors and high-efficiency electron optics, but low-concentration species with very low cross sections still present difficulties for analysis. The introduction of new X-ray anode materials or even synchrotron radiation providing photons with lower energies to enhance the sensitivity of the technique to particular bearing surface species is certainly possible. Finally, most surface science instruments are not designed to handle specimens having curved shapes such as bearing races. It is likely that custom sample handling mechanisms will have to be developed for particular applications.

Non-linear optical spectroscopies, such as second-harmonic generation and sum-frequency generation, are under development.[15] These techniques could provide surface and interfacial characterization at ambient pressures, avoiding the vacuum environments required for the electron spectroscopies reviewed in this text.

Finally, semiconductor-based microsensors could be used to monitor the health of a mechanism during operation. A micro-accelerometer package is currently flying on the Olympus spacecraft.[16] The accelerometers measure the vibration modes induced by the operation of mechanisms on the spacecraft. In the future, mass spectrometry could be used to investigate the gas-phase by-products of lubricant degradation that may occur during bearing tests of space applications. If such studies established gas-phase signatures to lubricant degradation, semiconductor-based chemical sensors could be tailored to monitor these ejected molecules. These sensors could be incorporated into the bearing mechanism as part of a health monitoring scheme to notify the user or an automated controller that can respond that additional lubrication is needed. The dawn of the "smart bearing" may be near.

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